

Air injection into concentrated maple sap during processing: impact on syrup composition and flavour

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Abstract

BACKGROUND: Air injection (AI) is a relatively new process used during maple sap thermal processing to increase the profitability of maple syrup production by increasing the production of more economically valuable light-coloured syrup. The effects of applying this technology in conjunction with existing practices employed to increase the efficiency of maple production, such as reverse osmosis (RO), are unknown. The main objective of this work was to investigate the effects of AI on syrup chemical composition and flavour when applied to maple sap concentrated by RO.

RESULTS: The chemical composition and flavour of syrup produced simultaneously with and without AI from a common source of maple sap concentrated by RO were compared. The chemical composition of maple syrup produced with AI was within ranges previously published for maple syrup. Syrup produced with AI was significantly lighter in colour than syrup produced without AI from the same sap concentrate ($P < 0.001$). Although syrup produced with AI contained fewer volatile flavour compounds and had a flavour distinguishable from that of syrup produced without AI from the same concentrated sap, the flavour properties of AI syrup were consistent with those of light-coloured maple syrup.

CONCLUSION: The results indicate that AI can be used in conjunction with RO to effectively increase the economic efficiency of maple syrup production without detrimental impacts on maple syrup properties.

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Keywords: maple syrup; air injection; maple sap concentrate; reverse osmosis; sugar

INTRODUCTION

Maple syrup is a liquid sugar produced from the sap collected from sugar maple (*Acer saccharum* Marsh.) and other trees of the genus *Acer*. Maple sap (approximately 2°Brix) is typically concentrated into maple syrup (66–67°Brix) by heating in a continuous process in evaporators specialised for maple production.¹ Chemical reactions that occur during this heating process yield the majority of the flavour and colour attributes characteristic of maple syrup.²

In addition to standard maple evaporators, many maple producers use auxiliary equipment to increase the efficiency and profitability of maple syrup production. For example, maple sap can be concentrated from 2 to 8–12°Brix by reverse osmosis (RO) prior to concentration in the evaporator.¹ This greatly reduces the amount of water that must be removed by heat-driven evaporation and thus reduces the amount of time and evaporator fuel required to process sap into syrup. This can substantially reduce the overall cost of producing maple syrup.¹

Another auxiliary device employed by some producers to increase the profitability of maple syrup production is air injection (AI), in which filtered ambient air is forced into the boiling sap in the evaporator through perforated stainless steel pipes placed in the evaporator pans. This process often results in the production of lighter-coloured syrup than what would have been produced without its application.³ While a maple producer will usually

produce syrup ranging from very light to very dark during a single production season, light-coloured maple syrup generally has greater economic value than dark-coloured syrup.² AI can thus increase the overall value of a producer's maple crop by increasing the proportion of light-coloured syrup produced.

Previous work has demonstrated that, when applied to unconcentrated ('raw') maple sap, AI produces lighter-coloured syrup with fewer volatile flavour compounds but does not otherwise substantially impact the bulk chemical composition of maple syrup.³ However, the effects of AI on syrup chemistry and flavour when used in conjunction with sap preconcentration by RO have not been investigated. As the majority of the

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chemical reactions responsible for maple syrup colour and flavour development occur during heating in the evaporator,² the combination of a technique that reduces sap residence time in the evaporator with one that produces lighter-coloured syrup has the potential to significantly impact the chemical and flavour properties of the maple syrup produced. Thus the objective of this project was to investigate the effects of AI on syrup chemistry and flavour when applied to maple sap preconcentrated by RO.

EXPERIMENTAL

Maple syrup production

Maple syrup production was conducted at the Maple Production Research Facility at the University of Vermont Proctor Maple Research Center in Underhill Center, Vermont, USA. Maple syrup was produced using two oil-fuelled maple evaporators (Dallaire Model Deluxe, size 0.9 m × 3 m, Les Équipements d'Érablière CDL, Saint-Lazare, Québec, Canada). Both evaporators were equipped with automatic sap level regulation and syrup draw-off devices and were configured to process sap concentrate as similarly as possible, with equal liquid depths and oil burner and exhaust draft settings. One evaporator (AI) was equipped with a standard AI system (Les Équipements d'Érablière CDL), while the other evaporator (control) functioned as a control treatment.

Maple syrup production experiments were repeated on seven days over the span of the 2007 maple production season. During each repetition of the experiment, maple syrup was produced simultaneously in the two evaporators while both were supplied from a common tank with sugar maple sap that had been concentrated to 8°Brix by RO ('concentrate') using a Springtech 1600 RO unit (Leader Evaporator, St Albans, VT, USA) equipped with a PVD-1 membrane (Hydranautics, Oceanside, CA, USA). The evaporators were started simultaneously and allowed to process the concentrate for at least 1 h beyond first syrup production. The AI system was located throughout the AI evaporator except within 40 cm of the syrup draw-off valve. The system was operated continuously throughout each experiment, with filtered ambient air supplied at a rate of approximately 2.8 m³ min⁻¹.

At the conclusion of each repetition, all syrup produced by each evaporator was filtered to remove suspended solids, adjusted to the correct density (Vermont standard minimum, 66.9°Brix) and subsamples were packaged for subsequent analyses.

Seven pairs of maple syrup samples were produced by the conclusion of the experiment. Each pair consisted of one syrup sample produced with (AI) and one produced without (control) AI simultaneously from the same maple sap concentrate.

Analyses

The colour, pH, conductivity and loose scale, inorganic mineral, nitrogen, carbohydrate and volatile flavour compound contents were determined for each syrup sample produced during the experiments. The thickness of mineral scale deposited on the evaporator pans from each treatment was also determined.

Colour

Maple syrup colour was determined by measuring the percentage of light transmittance at 560 nm with a Spectronic Genesys 8 UV–visible spectrophotometer (Thermo Electron, Waltham, MA, USA) using glycerol as a 100% transmittance standard.

Conductivity and pH

Conductivity (μS cm⁻¹) and pH were determined with an Accumet XL60 meter (Fisher Scientific, Fair Lawn, NY, USA).

Inorganic minerals and nitrogen

To determine the composition of inorganic mineral elements in each syrup sample, 0.5 g of each sample was digested with 10 mL of concentrated nitric acid for 15 min at 190 °C and 2.1 MPa pressure. Digested samples were then analysed for aluminium, calcium, copper, iron, magnesium, manganese, phosphorus, potassium, sodium, sulfur and zinc contents (mg kg⁻¹) by inductively coupled plasma atomic emission spectroscopy with a PlasmaSpec 2.5 instrument (Leeman Labs, Hudson, NH, USA).

The nitrogen content (g kg⁻¹) of each sample was determined with a Thermo Electron Flash EA 1112 Series NC elemental analyser (Thermo Finnigan Italia SpA, Rodana, Milan, Italy).

Carbohydrates

The sucrose, glucose and fructose contents (g kg⁻¹) of each syrup sample were determined by high-performance liquid chromatography (HPLC) using a 1525 binary pump and a 2410 refractive index detector (Waters, Milford, MA, USA). An Aminex HPX-87K column (300 mm × 7.8 mm, Bio-Rad, Hercules, CA, USA) was used at 75 °C with a mobile phase of 0.2 mmol L⁻¹ potassium phosphate at a rate of 0.6 mL min⁻¹. Glucose and fructose values were summed to calculate the total invert sugar content of each sample.

Volatile flavour compounds

The composition of volatile flavour compounds in each syrup sample was determined by an automated solid phase microextraction (SPME) method modified and adapted for maple syrup from one previously developed for sugar.⁴ Syrup samples (6 mL) were pipetted into pre-baked 10 mL vials. The vials were sealed with Teflon-lined silicon screw caps (Supelco, Inc., St Louis, MO, USA) and analysed by gas chromatography/time-of-flight mass spectrometry (GC/TOF-MS) using a Combi-PAL autosampler (Leap Technologies, Carrboro, NC, USA), an Agilent 6890 gas chromatograph (GC) (Agilent, Wilmington, DE, USA) and a Pegasus III time-of-flight mass spectrometer (TOF-MS) (Leco Corp., St Joseph, MI, USA). An empty vial was analysed as a blank.

Volatiles were extracted using a 1 cm 50/30 μm divinylbenzene/carboxen/polydimethylsiloxane fibre (Supelco, Inc.) chosen because of its wider range of polarity and its affinity for polar aromatic compounds. Following a 2 min heating period, extraction took place at 65 °C for 40 min. Samples were continuously agitated at 750 rpm during heating and at 250 rpm during extraction, with the agitation direction being reversed every 10 s. After extraction the fibre was desorbed in the GC injection port operated in splitless mode for 1 min at 270 °C. Prior to reuse the fibre was baked in a stream of helium at the same temperature in a fibre cleaning oven for 4 min to prevent carryover.

The GC was equipped with a 5% phenyl/95% dimethylpolysiloxane column (30 m × 0.25 mm, 0.25 μm, Model DB-5, Agilent) operated in constant flow mode with a helium velocity of 40 cm s⁻¹. The oven temperature was held at 35 °C for 1 min, then increased at a rate of 5 °C min⁻¹ to 180 °C, then at 20 °C min⁻¹ to 270 °C and held for 5.5 min. The GC interfaced to the TOF-MS through a 250 °C transfer line. The MS was scanned from 33 to 400 amu at 20 full spectra s⁻¹. The ion source temperature was held at 200 °C.

MS data were acquired and analysed by Chroma-TOF software (Leco Corp.). Compound identification was made by library match

with the Palisade Complete 600K library (Palisade Corporation, Ithaca, NY, USA). The total amount of volatile flavour compounds detected as well as the total composition of pyrazines and two compounds specifically associated with maple flavour, 2-hydroxy-3-methyl-2-cyclopenten-1-one and 3-ethyl-2-hydroxy-2-cyclopenten-1-one,^{5,6} were examined in each syrup sample. Relative quantities of volatile flavour compounds in each sample were expressed as peak area counts. Peak area counts for compounds identified with this method are considered semi-quantitative⁴ and can be used to express and compare relative quantities of compounds identified in the syrup samples analysed.

Loose scale

Prior to bulk filtration a small subsample of syrup from each treatment was passed through an individual preweighed, synthetic syrup filter (Leader Evaporator) to collect the loose mineral scale suspended in the syrup. The filters were rinsed with water to remove sugars, dried and reweighed in order to calculate the quantity of loose scale produced kg⁻¹ syrup in each treatment.

Scale deposition

At the conclusion of all experiments the evaporator pans were drained, rinsed with water and dried. The thickness (µm) of mineral scale deposited on the surface of the evaporator pans from each treatment was measured with a PosiTest DFT Combo ultrasonic coating thickness gauge (DeFelsko, Ogdensburg, NY, USA). Each set of evaporator pans was divided into six sections. Scale thickness measurements were collected at 3 cm intervals within each of these sections and used to calculate the mean scale thickness for each of the six sections of the evaporator pans for each treatment.

Sensory evaluation

Triangle tests were conducted to test for overall differences in the flavour of syrup produced simultaneously with and without AI using the same maple sap concentrate for each of the pairs of syrup produced on the seven days the experiment was run. Tests were performed following the procedures described by Meilgaard *et al.*⁷ Fifteen adult panellists with experience in tasting and grading maple syrup were selected. Panellists were separated by cardboard partitions under fluorescent light during administration of the test. The sample presentation order was randomised for each panellist, and opaque sample bottles were used to eliminate any influence of syrup colour on the panellists' perceptions. Pairs were considered different ($P < 0.05$) if nine of the 15 panellists positively identified the odd sample.⁷

Statistical analyses

All data were analysed using SAS Version 8e software (SAS Institute, Cary, NC, USA). Statistical assumptions of normality were verified using Shapiro–Wilks tests. For each parameter a paired Student's *t* test was used to test the hypothesis that means were equal between syrup produced with (AI) and without (control) AI using the same maple sap concentrate. Nonparametric Wilcoxon signed rank tests were used for populations when the assumption of normality was not met.

To evaluate the effects of AI on mineral scale deposition, mean scale thickness measurements were calculated for each section of the AI and control evaporator pans. A Wilcoxon signed rank test was used to test the hypothesis that the thickness of scale deposition was equal in the control evaporator and the evaporator equipped with AI.

RESULTS AND DISCUSSION

Syrup made with AI was significantly lighter in colour than control syrup simultaneously made without AI from the same maple sap concentrate (Table 1). This is consistent with results observed in experiments using AI with raw (unconcentrated) sap³ and indicates that using AI during sap processing with raw sap or RO concentrate results in maple syrup with significantly less colour than syrup made without AI. AI syrup also contained significantly fewer volatile flavour compounds than control syrup, though only at a marginal ($P < 0.08$) level (Table 1). In addition, the quantity of two compounds associated with maple flavour, 2-hydroxy-3-methyl-2-cyclopenten-1-one and 3-ethyl-2-hydroxy-2-cyclopenten-1-one,^{5,6} was significantly lower in syrup made with AI than in control syrup (Table 1). It is important to note, however, that these are only two of many compounds important to maple flavour.² These results are consistent with those obtained using AI with raw maple sap³ and suggest that using AI results in syrup with fewer flavour and aroma compounds. Given the impact of AI on syrup colour, this result is not unexpected, as many of the chemical processes that result in colour development during sap processing into syrup are also those that result in the formation of flavour and aroma compounds.^{2,8} These effects on flavour are consistent with expectations, as lighter-coloured syrups typically contain fewer flavour compounds than darker-coloured syrups.² Interestingly, AI syrup contained significantly greater quantities of pyrazines than control syrup (Table 1). This contrasts with observations that AI generally produces syrup with fewer flavour compounds, and it suggests that AI's effects on maple syrup flavour may be quite complex.

AI also affected the carbohydrate composition of maple syrup. AI and control syrup contained similar quantities of sucrose, the dominant carbohydrate in maple syrup;² however, syrup made with AI contained significantly greater quantities of glucose, fructose and total invert sugar than control syrup made simultaneously without AI using the same concentrate. Similar effects on carbohydrate composition were observed in experiments using AI with raw maple sap.³ These effects may be related to the reduced quantities of colour and flavour compounds observed in syrup made with AI, possibly reflecting that fewer reducing sugar precursors are being utilised in non-enzymatic browning reactions. Although differences were observed, the quantities of these carbohydrates in AI syrup were within ranges typically reported for maple syrup.^{2,9,10}

The pH and conductivity of syrup produced with AI did not differ significantly from those of syrup produced without AI (Table 1). AI also did not significantly affect the composition of most inorganic elements in maple syrup. AI and control syrup contained similar quantities of calcium, copper, iron, phosphorus, potassium, sodium, sulfur and zinc (Table 1). AI syrup did contain slightly, but significantly, greater quantities of aluminium, magnesium, manganese and nitrogen than control syrup made from the same concentrate, suggesting that the physical agitation caused by AI may prevent some mineral precipitation. Despite these differences, the quantities of inorganic elements found in syrup made with AI were all within the ranges typically reported for maple syrup.^{2,9,10} These results are also consistent with those obtained in experiments with AI and raw maple sap.³

During the evaporation process, mineral precipitates form in maple syrup. Some remain suspended in the syrup (termed 'sugar sand') and some are deposited as scale ('nitre') on the surfaces of evaporator pans. Both are considered a nuisance to maple producers and can reduce the efficiency of maple syrup

Table 1. Chemical composition (mean \pm standard error of mean) of maple syrup produced with (AI) and without (control) air injection using a common source of maple sap concentrate

Parameter measured	Treatment		Probability level <i>P</i>
	AI	Control	
Light transmittance (%)	73.8 \pm 6.1	35.4 \pm 4.7	0.001
Conductivity ($\mu\text{S cm}^{-1}$)	136.3 \pm 5.0	133.1 \pm 8.5	0.638
pH	6.7 \pm 0.5	6.7 \pm 0.8	0.476
Aluminium (mg kg^{-1})	77.1 \pm 12.3	50.3 \pm 9.5	0.056
Calcium (mg kg^{-1})	1188.7 \pm 90.4	1157.0 \pm 76.1	0.219 ^a
Copper (mg kg^{-1})	60.6 \pm 9.5	45.2 \pm 10.4	0.162
Iron (mg kg^{-1})	7.9 \pm 1.2	7.1 \pm 1.6	0.531
Magnesium (mg kg^{-1})	196.6 \pm 10.6	148.7 \pm 8.6	0.016 ^a
Manganese (mg kg^{-1})	21.2 \pm 5.4	15.5 \pm 4.8	0.047 ^a
Nitrogen (g kg^{-1})	0.27 \pm 0.05	0.22 \pm 0.06	0.047 ^a
Phosphorus (mg kg^{-1})	69.7 \pm 4.1	63.4 \pm 5.1	0.126
Potassium (mg kg^{-1})	1685.1 \pm 63.8	1586.5 \pm 102.5	0.688 ^a
Sodium (mg kg^{-1})	10.2 \pm 0.9	9.8 \pm 1.9	0.802
Sulfur (mg kg^{-1})	91.6 \pm 38.0	81.1 \pm 17.4	0.747
Zinc (mg kg^{-1})	4.9 \pm 0.4	5.0 \pm 0.8	0.888
Sucrose (g kg^{-1})	644.4 \pm 7.4	644.0 \pm 7.5	0.934
Glucose (g kg^{-1})	4.2 \pm 1.0	3.3 \pm 0.7	0.016 ^a
Fructose (g kg^{-1})	4.0 \pm 0.9	3.5 \pm 0.7	0.016 ^a
Total invert sugar (g kg^{-1})	8.2 \pm 1.9	6.8 \pm 1.4	0.016 ^a
Loose scale (g kg^{-1})	0.6 \pm 0.1	0.6 \pm 0.1	0.837
Volatile flavour compounds (peak area count)	(5.9 \pm 0.96) $\times 10^7$	(8.3 \pm 1.7) $\times 10^7$	0.077
Maple flavour compounds (peak area count)	(6.0 \pm 2.8) $\times 10^5$	(4.4 \pm 1.2) $\times 10^6$	0.007
Total pyrazines (peak area count)	(9.7 \pm 2.8) $\times 10^6$	(4.2 \pm 1.8) $\times 10^6$	0.011

P values are for paired Student's *t* tests comparing mean values for AI and control syrup (*n* = 7).

Maple flavour compounds represent sum quantities of 2-hydroxy-3-methyl-2-cyclopenten-1-one and 3-ethyl-2-hydroxy-2-cyclopenten-1-one.

^a Mean comparisons made with Wilcoxon signed rank tests.

production. Loose mineral scale suspended in syrup rapidly clogs filters during syrup production, while scale deposited on pans reduces heat transfer, increases the possibility of pan scorching, can impart an off-flavour to the syrup and necessitates shutting down the system for periodic cleaning.¹ Anecdotally, AI is reported to reduce both loose scale development and scale deposition on pans, resulting in an increase in the overall efficiency of maple syrup production. However, the amount of loose scale filtered from syrup in this study was equal in AI and corresponding control syrup (Table 1). In addition, the thickness of scale deposited on evaporator pans did not differ significantly between the treatments (Table 2). These results are similar to those obtained in experiments with AI using raw maple sap³ and indicate that AI does not reduce the production or deposition of mineral scale when using maple sap concentrated by RO.

Results from sensory evaluations indicate that panellists perceived a general difference in the flavour of syrup produced with and without AI from the same maple sap concentrate for each of the five pairs of syrup samples produced earliest in the maple production season (Table 3). Panellists did not detect differences in the syrup produced with and without AI from the same concentrate on either of the final two dates of the production season, suggesting that AI may have a smaller impact later in the maple production season. Although triangle tests do not indicate the nature of the perceived difference in flavour, these results suggest that AI does impact maple syrup flavour and are consistent with the lower amount of volatile flavour

Table 2. Scale deposition (mean \pm standard error) on evaporator pans used to produce maple syrup with (AI) and without (control) air injection using a common source of maple sap concentrate

Parameter measured	Treatment		<i>P</i>
	AI	Control	
Scale thickness (μm)	9.9 \pm 3.5	8.3 \pm 3.4	1.00

The *P* value is for a paired Wilcoxon signed rank test comparing mean scale thicknesses in six sections of the AI and control evaporators.

compounds observed in AI syrup *versus* control syrup. However, these results are also consistent with typical expectations of syrup flavour. Lighter-coloured syrups characteristically contain fewer flavour compounds and are evaluated as having a flavour distinctly different from that of darker-coloured syrups.^{2,11} Thus the general differences in flavour observed in this study are expected given the lighter colour of syrup produced with AI relative to the darker colour of control syrup made from the same concentrate.

The mechanism through which AI exerts its effects on maple syrup was not specifically investigated in this study. Liquid temperatures in the AI evaporator were an average of 8.0 °C lower (standard error of mean 2.7 °C, range 0.5–16.0 °C) than the liquid temperatures in corresponding locations in the control evaporator (data not shown). It is reasonable to hypothesise that

Table 3. Number of correct responses (odd sample positively identified) in triangle tests conducted with 15 panellists to determine if differences in flavour were detectable between pairs of syrup samples produced with and without air injection simultaneously from the same maple sap concentrate on seven different dates during the 2007 maple production season

Production date	Number of correct responses
15 March	13*
16 March	10*
23 March	9*
28 March	9*
29 March	9*
3 April	5
13 April	5

* A statistically significant difference ($P < 0.05$) was detected between the pair of samples.

some of the observed effects of AI on maple syrup, including higher invert sugar levels and fewer colour and flavour compounds, could be the result of reductions in non-enzymatic browning reactions due to lower processing temperatures. However, it is possible that AI may also impact maple syrup properties through other mechanisms, including through the effects of chemical oxidation or mechanical agitation potentially caused by the AI process. Further experiments would be necessary in order to precisely determine the mechanisms through which AI yields effects on maple syrup properties.

CONCLUSIONS

When processing maple sap concentrated by RO, AI produced lighter-coloured syrup than control syrup made without AI. However, the use of AI did not decrease the amount of scale precipitated or deposited on evaporator pan surfaces. These results indicate that AI can be used in conjunction with RO to increase the economic efficiency of maple production by increasing the production of more economically valuable light-coloured syrup, but not through reductions in scale development. Using AI with sap concentrated by RO did not substantially alter the chemical composition of maple syrup. Although it is possible that AI could affect the composition of other constituents, such as phenolic compounds, not examined in this study, the results suggest that using AI with concentrated sap produces syrup with a composition consistent with that of pure maple syrup. The observed impacts on syrup flavour were consistent with expectations of lighter-coloured maple syrup and indicate that AI produces light-coloured

syrup with flavour properties generally consistent with those of light-coloured syrup. In conclusion, these results indicate that AI can be used in conjunction with RO to safely and effectively increase the economic efficiency of maple syrup production.

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